**BRITISH STANDARD**

**BS EN 725-1 : 1997**

# Advanced technical ceramics — Methods of test for ceramic powders

## Part 1. Determination of impurities in alumina

The European Standard EN 725-1 : 1997 has the status of a British Standard

ICS 81.060.99

# Committees responsible for this British Standard

The preparation of this British Standard was entrusted to Technical Committee RPI/13, Advanced technical ceramics, upon which the following bodies were represented:

AEA Technology
Aluminium Federation
British Ceramic Research Ltd.
British Industrial Ceramic Manufacturers' Association
Department of Trade and Industry (National Physical Laboratory)
Flat Glass Manufacturers' Association
GAMBICA (BEAMA Ltd.)
Institute of Refractories Engineers
Ministry of Defence
Refractories Association of Great Britain
Society of British Aerospace Companies Ltd.
University of Manchester

This British Standard, having been prepared under the direction of the Sector Board for Materials and Chemicals, was published under the authority of the Standards Board and comes into effect on
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**Amendments issued since publication**

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# Contents

# National foreword

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EN 725-1 : 1997 was produced as a result of international discussions in which the United Kingdom took an active part.

EN 725-1 : 1997 has been approved by CEN member bodies under the weighted voting procedures introduced in 1988 to coincide with the introduction of 'New Approach' Directives from the Commisssion of the European Community.

## Cross-references

| **Publication referred to** | **Corresponding British Standard** |
|---|---|
| ISO 3696 | BS 3978 : 1995 *Water for analytical laboratory use — Specification and test methods* |
| ISO 5725 | BS 5497 *Precision of test methods*<br>Part 1 : 1987 *Guide for the determination of repeatability and reproducibility for a standard test method by inter-laboratory tests* |

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## Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, the EN title page, pages 2 to 6, an inside back cover and a back cover.

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# EN 725-1

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English version

# Advanced technical ceramics — Methods of test for ceramic powders — Part 1: Determination of impurities in alumina

Céramiques techniques avancées — Méthodes d'essai pour poudres céramiques — Partie 1: Détermination des impuretés dans l'alumine

Hochleistungskeramik — Prüfverfahren für keramische Pulver — Teil 1: Bestimmung von Verunreinigungen in Aluminiumoxidpulver

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Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

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European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

**Central Secretariat: rue de Stassart 36, B-1050 Brussels**

Ref. No. EN 725-1 : 1997 E

## Foreword

This European Standard has been prepared by Technical Committee CEN/TC 184, Advanced technical ceramics, the secretariat of which is held by BSI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by September 1997, and conflicting national standards shall be withdrawn at the latest by September 1997.

EN 725 consists of 11 parts:

Part 1 : *Determination of impurities in alumina*
Part 2 : *Determination of impurities in barium titanate (ENV)*
Part 3 : *Determination of oxygen content of non-oxides by thermal extraction*
Part 4 : *Determination of oxygen content of non-oxides by XRF analysis (ENV)*
Part 5 : *Determination of particle size distribution*
Part 6 : *Determination of specific area*
Part 7 : *Determination of absolute density*
Part 8 : *Determination of tapped bulk density*
Part 9 : *Determination of untamped bulk density*
Part 10 : *Determination of compaction properties*
Part 11 : *Determination of reactivity on sintering (ENV)*

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## Contents

## 1 Scope

This part of EN 725 specifies methods for the determination of elements of sodium, potassium, iron, silicon, calcium and magnesium present as impurities in alumina using atomic absorption (AAS) or inductively coupled plasma (ICP) instruments. For each element present as impurities, the methods are applicable to the following ranges, calculated as oxides:

| | |
|---|---|
| Sodium oxide | 20 ppm to 6000 ppm |
| Potassium oxide | 20 ppm to 100 ppm |
| Ferric oxide | 20 ppm to 300 ppm |
| Silica | 50 ppm to 2000 ppm |
| Calcium oxide | 20 ppm to 700 ppm |
| Magnesium oxide | 5 ppm to 1000 ppm |

## 2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and in the publications listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

| | |
|---|---|
| ECSC/CI 9 | *Chemical analysis of ferrous materials — Operational guidelines for the application of flame atomic absorption spectrometry in standard methods for the chemical analysis of iron and steel* |
| ISO 3696 | *Water for analytical laboratory use — Specification and test methods* |
| ISO 5725 | *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests* |
| ISO/DIS 13527 | *Chemical analysis of ferrous materials — Guidelines on the use of inductively coupled plasma atomic emission spectroscopy* |

## 3 Principle

The test sample is decomposed by using either a fusion method or an acid dissolution method. The acid dissolution method cannot be used for the determination of silicon. The solution is transferred to a volumetric flask and diluted to a known volume, and the elements are determined by AAS or ICP (see clause **1**).

## 4 Reagents

### 4.1 General

During the analysis, use only reagents and calibration solutions of at least 99,99 % purity and only distilled water or water of equivalent purity (see ISO 3696).

### 4.2 Reagents for fusion

**4.2.1** *Lithium carbonate*, $Li_2CO_3$

**4.2.2** *Potassium carbonate*, $K_2CO_3$

**4.2.3** *Boric acid*, $H_3BO_3$

**4.2.4** *Sulfuric acid*, $H_2SO_4$, ($\rho_{20}$ = 1,84 g/ml)

**4.2.5** *Lithium metaborate*, $LiBO_2$

**4.2.6** *Nitric acid*, $HNO_3$, ($\rho_{20}$ = 1,33 g/ml)

**4.2.7** *Phosphoric acid*, $H_3PO_4$, ($\rho_{20}$ = 1,78 g/ml)

### 4.3 Reagents for acid dissolution

**4.3.1** *Sulfuric acid-phosphoric acid mixture (A)*

Pour 500 ml of phosphoric acid ($\rho_{20}$ = 1,78 g/ml) into 500 ml of sulfuric acid ($\rho_{20}$ = 1,84 g/ml)

**4.3.2** *Sulfuric acid-phosphoric acid mixture (B)*

Pour 700 ml of phosphoric acid ($r_{20}$ = 1,78 g/ml) into 300 ml of sulfuric acid ($\rho_{20}$ = 1,84 g/ml).

### 4.4 Reagents for calibration

**4.4.1** *Pure alumina*, of very low and known impurity levels.

**4.4.2** *Sodium*, commercial solution or solution obtained by dissolution of pure chemical compound, concentration 1 g/l.

**4.4.3** *Potassium*, commercial solution or solution obtained by dissolution of pure chemical compound, concentration 1 g/l.

**4.4.4** *Iron (ferric)*, commercial solution or solution obtained by dissolution of pure chemical compound, concentration 1 g/l.

**4.4.5** *Silicon*, commercial solution or solution obtained by dissolution of pure chemical compound, concentration 1 g/l.

**4.4.6** *Calcium*, commercial solution or solution obtained by dissolution of pure chemical compound, concentration 1 g/l.

**4.4.7** *Magnesium*, commercial solution or solution obtained by dissolution of pure chemical compound, concentration 1 g/l.

## 5 Apparatus

**5.1** *Platinum crucible*, with a capacity of at least 50 ml.

**5.2** *Gold-platinum crucible*, with a capacity of at least 50 ml.

**5.3** *Vitreous carbon crucible*, with a capacity of at least 50 ml with lid and heating device.

**5.4** *Muffle furnace*, suitable for operation in the range of 1000 °C to 1200 °C.

**5.5** *Magnetic stirrer*, with heating.

**5.6** *Atomic absorption spectrometer and/or inductively coupled plasma spectrometer*, in accordance with ECSC/CI 9 or ISO 13527 respectively.

**5.7** *Laboratory glassware.*

## 6 Test sample

Use samples of approximately :

- 2 g for decomposition by fusion;
- 1 g for decomposition by acid dissolution.

Weigh them to 0,0005 g.

## 7 Decomposition of the test sample

### 7.1 General

Dissolve either by a fusion method (see **7.2** to **7.4**) or an acid dissolution method (see **7.5** and **7.6**)

### 7.2 Fusion 1

In a platinum crucible (see **5.1**) weigh 1,5 g $Li_2CO_3$ (see **4.2.1**) 5 g $K_2CO_3$ (see **4.2.2**) and 2,5 g $H_3BO_3$ (see **4.3.3**). Add the test sample of approximately 2 g (see clause **6**) and mix intimately using a platinum spatula.

Place the crucible and contents into the muffle furnace (see **5.4**), maintained at 1050 °C ± 50 °C, for 30 min. Remove the crucible from the furnace and swirl the contents on the sides of the crucible, then allow to cool to room temperature.

Dissolve the fused product in a 400 ml beaker which contains 100 ml of water and 10 ml of sulfuric acid (see **4.2.4**). Place the beaker, covered with a watch glass, on a hot plate and heat to boiling. Maintain at boiling point to obtain a complete dissolution. Remove the beaker from the hot plate. Allow to cool.

Transfer quantitatively the solution into a 200 ml volumetric flask. This procedure allows for a concentration of alumina up to 8 g/l but if needed, a dilution to a higher volume is possible.

Allow to cool to room temperature and make up to the mark.

### 7.3 Fusion 2

In a platinum crucible (see **5.1**) weigh 4 g of $LiBO_2$ (see **4.2.5**) and 1 g of test sample. Mix intimately using a platinum spatula.

Place the crucible and contents into the muffle furnace (see **5.4**) maintained at 1150 °C ± 50 °C for 30 min (after the first 15 min, swirl the contents of the crucible for a few seconds). Remove the crucible from the furnace and dip its base in water at ambient temperature (this procedure allows easy removal of the bead from the crucible). To prevent sticking of melt in the crucible, either use a new crucible or, with an old one, immerse it in the solution. Place the bead into a 400 ml beaker which contains 80 ml of water and 20 ml of nitric acid (see **4.2.6**).

Place the beaker, covered with a watch glass on a magnetic stirrer with heating stirrer and maintain the agitation at approximately 80 °C ± 10 °C until complete dissolution. Remove the beaker from the stirrer and allow to cool. Transfer quantitatively the solution into a 200 ml volumetric flask. This procedure allows for a concentration of alumina up to 8 g/l but if needed, a dilution to a higher volume is possible. Allow to cool to room temperature and make up to the mark.

### 7.4 Fusion 3

In a gold-platinum crucible (see **5.2**), weigh 2,2 g $Li_2CO_3$ (see **4.2.1**) and 5,5 g $H_3BO_3$ (see **4.2.3**). Add the test sample (see clause **6**). Mix intimately using a platinum spatula.

Place the crucible and contents into the muffle furnace (see **5.4**), maintained at 1100 °C ± 50 °C for 30 min. Remove the crucible from the furnace and swirl the contents on the sides of the crucible, then allow to cool to room temperature. Dissolve the fused product in a 400 ml beaker which contains 100 ml of water and 20 ml of sulfuric acid (see **4.2.4**).

Place the beaker, covered with a watch glass, on a hot plate and heat to boiling. Maintain at boiling point to obtain complete dissolution. Remove the beaker from the hot plate. Allow to cool. Transfer quantitatively the solution into a 200 ml volumetric flask. This procedure allows for a concentration of alumina up to 8 g/l, but if needed a dilution to a higher volume is possible. Allow to cool to room temperature and make up to the mark.

### 7.5 Acid dissolution 1

Weigh the test sample (see clause **6**) into a vitreous carbon crucible (see **5.3**). Add carefully 15 ml of sulfuric acid-phosphoric acid mixture (see **4.3.1**). Cover with a lid. Put the crucible with the lid into the heating device and maintain at boiling for 20 min. Remove the crucible from the heating device and allow to cool to room temperature.

Transfer quantitatively the contents into a 100 ml volumetric flask which contains 30 ml of water. Rinse the crucible and the lid with distilled water into the flask and after cooling, make up to the mark with water.

### 7.6 Acid dissolution 2

Weigh the test sample (see clause **6**) into a gold-platinum crucible (see **5.2**). Add carefully 12 ml of sulfuric acid-phosphoric mixture (see **4.3.2**) and cover with a lid. Put the crucible with the lid on to the hot plate (see **5.5**) and maintain at boiling for 12 min. Remove the crucible from the heating device and allow to cool.

Transfer quantitatively the contents into a 100 ml volumetric flask which contains 30 ml of water. Rinse the crucible and the lid with distilled water into the flask and after cooling, make up to the mark with distilled water.

## 8 Calibration graph

### 8.1 General

The optimum calibration graph is obtained using calibration solutions whose concentrations are compatible both with the analytical method (AAS or ICP) and with the impurity concentrations in the sample.

The following procedure is given as an example.

### 8.2 Fusion

Prepare five decompositions of pure alumina (see **4.4.1**) in accordance with **7.2**, **7.3** or **7.4**. Transfer into five 200 ml volumetric flasks and dilute to 150 ml with water.

Add the quantities of solutions (see **4.4.2** and **4.4.4** to **4.4.7** for fusion 1 or **4.4.2** to **4.4.7** for fusion 2 and fusion 3) indicated in table 1. Make up to the mark with water.

**Table 1. Quantities of solutions for fusion**

| Elements | 1 | 2 | 3 | 4 | 5 |
|---|---|---|---|---|---|
| Na | 0 µl | 1 ml | 2 ml | 4 ml | 6 ml |
| K | 0 µl | 100 µl | 200 µl | 300 µl | 400 µl |
| Ca | 0 µl | 250 µl | 500 µl | 750 µl | 1000 µl |
| Fe | 0 µl | 100 µl | 200 µl | 300 µl | 400 µl |
| Si | 0 µl | 200 µl | 400 µl | 800 µl | 1600 µl |
| Mg | 0 µl | 250 µl | 500 µl | 750 µl | 1000 µl |

### 8.3 Acid dissolution

Prepare 5 dissolutions of pure alumina (see **4.4.1**) in accordance with **7.5** or **7.6**. Transfer into five 100 ml volumetric flasks and dilute to 50 ml with water. Add the quantities indicated in table 2. Make up to the mark with water.

**Table 2. Quantities of solutions for acid dissolution**

| Elements | 1 | 2 | 3 | 4 | 5 |
|---|---|---|---|---|---|
| Na | 0 µl | 500 µl | 1000 µl | 2000 µl | 3000 µl |
| K | 0 µl | 50 µl | 100 µl | 150 µl | 200 µl |
| Ca | 0 µl | 125 µl | 250 µl | 375 µl | 500 µl |
| Fe | 0 µl | 50 µl | 100 µl | 150 µl | 200 µl |
| Mg | 0 µl | 125 µl | 250 µl | 375 µl | 500 µl |

### 8.4 Drawing the calibration curve

#### 8.4.1 *Blank test*

Prepare a blank test in accordance with **8.2**, **8.3**, **8.4**, **8.5** or **8.6**, using the same quantities of all reagents as for dissolution of the test sample, but using pure alumina (see **4.4.1**) in place of the test sample.

#### 8.4.2 *Drawing the calibration curve*

With the calibration solution prepared according to **8.3**, curves can be drawn directly in ppm of impurity by using table 3 in which $b_i$ is the quantity of each oxide present as impurity in the solution obtained according to **8.4.1**.

**Table 3. Calibrations**

| Oxides | 1 | 2 | 3 | 4 | 5 |
|---|---|---|---|---|---|
| $Na_2O$ (ppm) | $b_1$ | 674 + $b_1$ | 1348 + $b_1$ | 2695 + $b_1$ | 4044 + $b_1$ |
| $K_2O$ (ppm) | $b_2$ | 60 + $b_2$ | 120 + $b_2$ | 180 + $b_2$ | 240 + $b_2$ |
| CaO (ppm) | $b_3$ | 175 + $b_3$ | 350 + $b_3$ | 525 + $b_3$ | 700 + $b_3$ |
| $Fe_2O_3$ (ppm) | $b_4$ | 71 + $b_4$ | 143 + $b_4$ | 214 + $b_4$ | 286 + $b_4$ |
| $SiO_2$ (ppm) | $b_5$ | 214 + $b_5$ | 428 + $b_5$ | 856 + $b_5$ | 1711 + $b_5$ |
| MgO (ppm) | $b_6$ | 207 + $b_6$ | 414 + $b_6$ | 622 + $b_6$ | 829 + $b_6$ |

## 9 Adjustment of the apparatus

### 9.1 Atomic absorption spectrometer
(see ECSC/CI9)

Follow the manufacturer's instructions for igniting and extinguishing the nitrous oxide-acetylene flame to avoid explosion, and ensure the safety screen is in place.

Set the wavelengths for the elements to be analyzed (see table 4) and adjust the apparatus so as to obtain maximum absorbance. Fit the correct burner and, in accordance with the manufacturer's instructions, light the flame. After 10 min preheating of the burner, adjust fuel and burner to obtain maximum absorbance while aspirating the highest calibration solution.

Aspirate water and set to give the zero absorbance, alternately aspirate the calibration solutions and water to establish that the absorbance reading is not drifting and draw the calibration graph.

### 9.2 Inductively coupled plasma spectrometer

(see ISO 13527)

Follow the manufacturer's instructions for igniting the plasma. Ensure the safety screen is in place.

The wavelengths in table 4 may be used for the analysis (according to analytical method and elements concentration, other wavelengths can also be used).

Wait until a stable signal is obtained. It is possible to use the A1 signal as an internal standard to improve the precision of the results.

**Table 4. Analysis lines**

| Elements | Atomic absorption (recommendations) | ICP (recommendations) |
|---|---|---|
| Na | 589,0 nm | 589,0 nm |
| K | 766,5 nm | 769,9 nm |
| Ca | 422,7 nm | 393,3 nm |
| Fe | 248,3 nm | 259,9 nm |
| Si | 251,6 nm | 251,6 nm |
| Mg | 285,2 nm | 279,5 nm |

## 10 Measurements

Aspirate the calibration solutions and the final test solutions in order of increasing concentrations. Aspirate water between each solution and record the readings when stable responses are obtained. Repeat the measurements at least twice more and calculate the average of the readings for each solution.

For each element, drawn the calibration curve by plotting the signal values of the calibration solution against the quantities of element oxide in ppm (see table 3). Convert the signal value of the test solution to ppm by mean of the calibration curves to obtain the raw result.

## 11 Expression of the results

Calculate the concentration of oxide in ppm by subtracting the from the raw result obtained according to clause **10**, the blank value $b_i$ (this value $b_i$ is the concentration read at the intersection of the calibration curve with concentration axis).

## 12 Accuracy

Examples of the reproducibility (*R*) and repeatability (*r*) of these measurements, determined in accordance with ISO 5725, using a standard reference alumina (NBS 699) are given in table 5.

**Table 5. Reproducibility and repeatability**

| Oxides | Results number | Mean (ppm) | *r* (ppm) | *R* (ppm) | Certified value (ppm) |
|---|---|---|---|---|---|
| $Na_2O$ | 55 | 5778 | 228 | 763 | 5900 ± 100 |
| $K_2O$ | 40 | 66 | 10 | 36 | 50 (not certified) |
| $Fe_2O_3$ | 48 | 132 | 23 | 38 | 130 ± 10 |
| $SiO_2$ | 43 | 112 | 26 | 121 | 140 ± 20 |
| CaO | 51 | 343 | 18 | 88 | 360 ± 20 |
| MgO | 32 | 5 | 2 | 3 | 6 ± 2 |

## 13 Test report

The test report shall include the following information:

a) name of testing establishment;

b) place and date of test, report identification, signatory;

c) reference to this European Standard (EN 725-1), including information on the method used (AAS or ICP);

d) details of the equipment used, if needed;

e) any information on the decomposition of the sample;

f) calibration procedure;

g) material type, manufacturing code, batch number;

h) relevant test parameters;

i) results of individual analyses;

j) mean results and standard deviation;

k) reproducibility and repeatability of the method;

l) comments about the test and test results.

# List of references

See national foreword.

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